

Robust UHTC for Passive Sharp Leading Edge Applications

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and Elizabeth J. Opila^b**

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Ultrahigh temperature ceramics have performed unreliably due to material flaws and attachment design. These deficiencies are brought to the fore by the low fracture toughness and thermal shock resistance of UHTCs. If these deficiencies are overcome, we are still faced with poor oxidation resistance as a limitation on UHTC applicability to reusable launch vehicles. We have been addressing the deficiencies of UHTCs for the past year via a small task at GRC that is part of the 3rd Gen TPS effort. Our focus is on composite constructions and functional grading to address the mechanical issues and on composition modification to address the oxidation issue. The approaches and progress will be reported.

a: QSS, Inc.

b: Cleveland State University

Outline

- Introduction
 - UHTC Background
 - Performance Issues for Leading Edges
- Robust UHTC: Objective and Approaches
- Oxidation Resistance Improvement
- Functional Gradient and Composite Materials
- Summary and Conclusions

Ultra High Temperature Ceramics

- Materials consisting of refractory metal borides, refractory metal carbides, silicon carbide, and carbon which have potential use temperatures limited by the melting point of the oxide scale.
 - HfO_2
 - ZrO_2
 - SiO_2

	Melting points	{	5073°F(2801°C)
			4904°F(2707°C)
			3142°F(1728°C), cristobalite
- ZrO_2 is not a highly protective oxide. Lifetimes based on ZrB_2 recession will be relatively short. 20 volume % SiC additions have been found to give lowest oxidation rates.

Clougherty, Pober, Kaufman, Trans Met Soc AIME 242, 1077 (1968).
Tripp, Davis, Graham, Cer Bull 52 [8] 612 (1973).
- Current Fabrication Approaches: hot pressing or chemical vapor infiltration

Potential Applications

- Inlets, nose tips, leading edges
- Satellite on-board propulsion system components
- Nozzle throats for divert and attitude control thrusters
- High performance short-life turbines

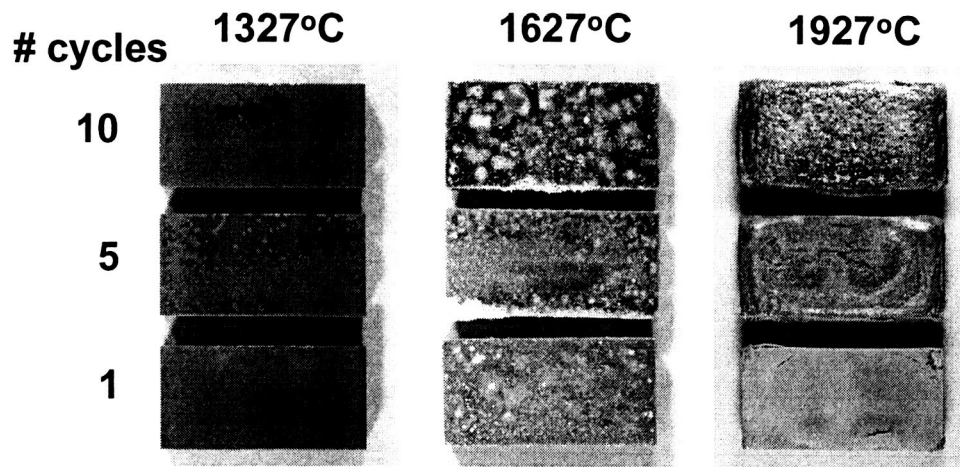
UHTC Lack Robustness

- UHTC have performed unreliably in ARC tunnels and flight due to material and attachment design flaws — ~~material flaws have been observed~~
- Oxidation resistance is unacceptable for reusable TPS leading edge applications
- Current UHTC materials are not reproducible and reliable
 - Partially intrinsic: low fracture toughness and strength
 - Partially extrinsic: process derived flaws
 - Thermal shock an area of concern

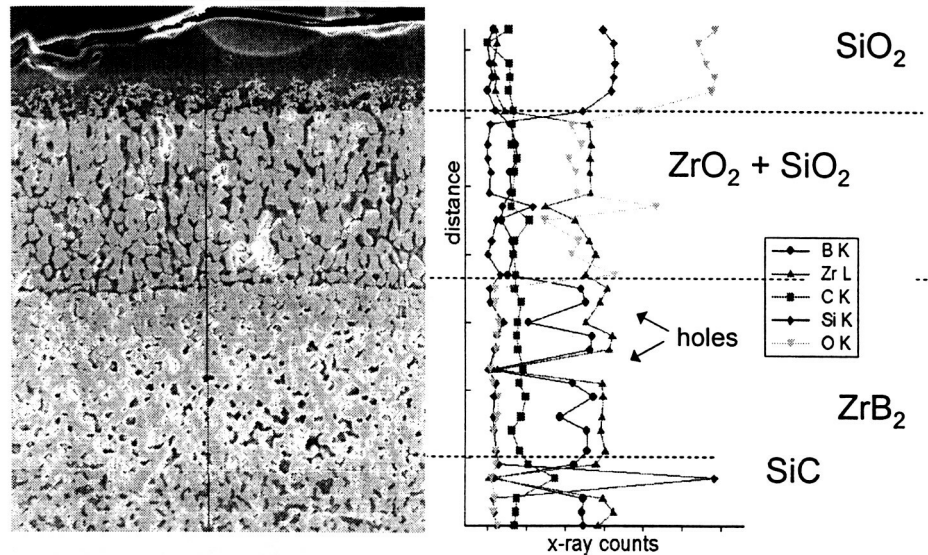
Soft Red d. this

ZrB₂/ 20 % SiC UHTC Oxidized in Air

10 Minute Cycles



SEM/EDS: ZrB₂ - SiC 1627°C, Air, Ten 10 min Cycles



Robust UHTC

- **Objectives**
 - Increase toughness and reliability
 - Improve oxidation resistance
- **Approach**
 - Alloying to improve oxidation resistance
 - Composites and functionally graded material (FGM)
 - Combine oxidation improvements with composite and FGM approaches

Oxidation Resistance

- Pure, dense ZrO_2 or HfO_2 scale impossible
 - Off stoichiometry
 - Porosity, cracking and spalling due to gaseous oxidation products and CTE mismatch
- Doped ZrO_2 or HfO_2
 - Limit oxygen transport via lattice vacancies
 - Still must deal with porosity, cracking, and spalling
 - Ta_2O_5 is the most practical based on melting point and formation of an intermediate ternary oxide with ZrO_2 and HfO_2
 - Potential sources of tantalum are:
 - TaB_2 or TaB : introduces B
 - TaC : introduces additional C
 - TaSi_2 or Ta_5Si_3 : introduces more Si + relatively low melting TaSi_x (~2400 °C)
- Just beginning to think about Re_2ZrO_7 or Re_2HfO_7

Properties of Relevant Compounds

Compound	Density	CTE, 10E6*cm/cmC	MP, C
B4C	2.5	6	2350
BN	2.25	3.8	3000(s)
HfB2	11.2	6.3	3250
HfO2	9.7	6.5	2810
Nb2O5	4.6	1	1460
NbB2	7.2	8	>2900(d)
NbC	7.8	7.2	3500
SiC	3.2	5.1	2500(d)
SiO2	2.7	3	1710
Ta2O5	8.7	2.4	1880
Ta5Si3	13.1	6.7	2460
TaB	14.3		3090
TaB2	12.6	8.2	3000(d)
TaC	14.5	7.1	3880
TaSi2	8.8	8.9	2200
TiB2	4.5	5.2	2980
TiC	4.9	8	3250
TiO2	4.3	9.5	1920
ZrB2	6.1	5.9	3040
ZrO2	5.8	8.1	2690

Additives
selected to
replace
diboride

History for Ta Additions

- Wuchina et al (NSWC) looked at TaB and TaC additions to pure HfC, HfN and HfB₂
 - Limited reporting of results
 - Denser oxide scale at 1500°C and 25% additive reported for HfB₂
- Talmy et al (NSWC) looked at up to 20% TaB₂ additions to ZrB₂ + 20%SiC
 - Only looked at furnace oxidation temperatures up to 1400°C
 - Significant improvements in oxidation resistance attributed to phase separation in the glass
 - Other mechanisms are possible

Mechanisms for ZrO₂ Protective Scale Enhancement via Ta₂O₅ Additions

- Ta₂O₅ as a glass modifier
 - Talmy et al
- Ta⁺⁵ as a dopant in ZrO₂ lattice
 - Fill O⁻² vacancies
 - Decrease O⁻² transport
- Ta₂O₅ as a major oxide scale constituent
 - Phase V
 - Low melting point

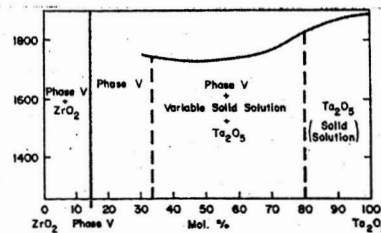
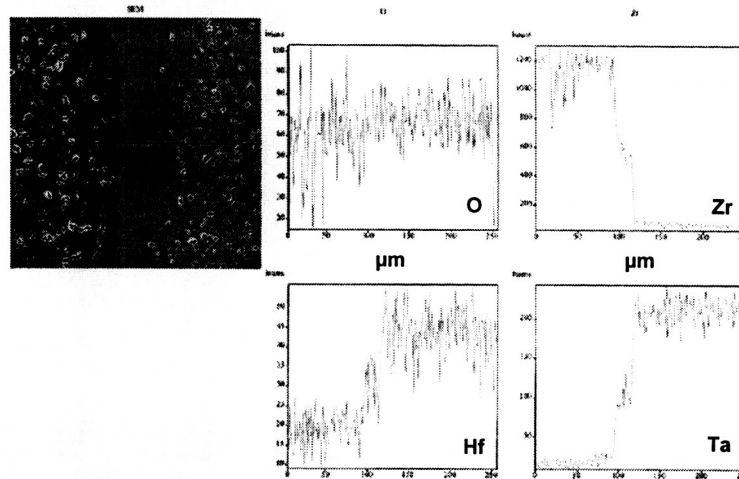


FIG. 374.—System Ta₂O₅-ZrO₂.

B. W. King, John Schultz, E. A. Durbin, and W. H. Duckworth, U. S. Atomic Energy Comm., BMI-1106, 15 (1956).

ZrO₂-Ta₂O₅ Diffusion Couple 1450°C, 1 h



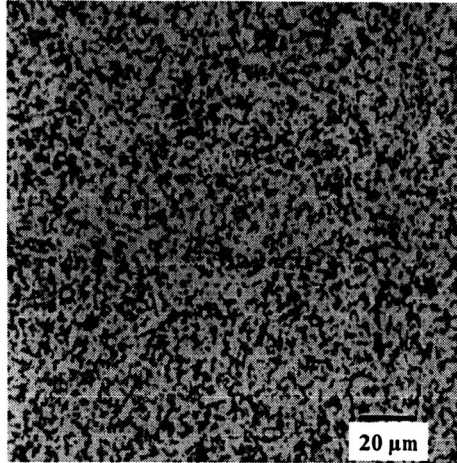
Intermediate phase formation and Ta solubility are evident

Processing

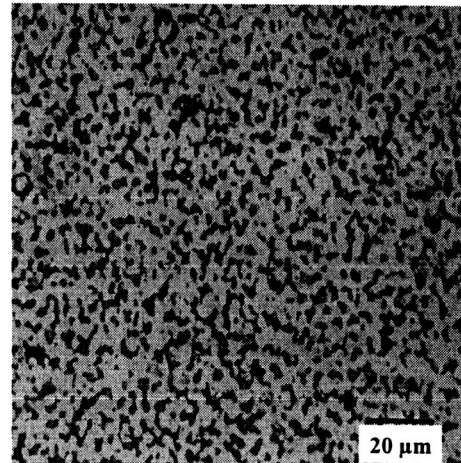
- **Baseline ZrB₂**
 - Plates hot pressed to > 95% average density at 2000°C, 10ksi, up to 2h, vac
- **Ball milling of mixed powders identified as preferred powder processing procedure**
 - One composition processed to 100% density at 1900°C, 10ksi, 2h, vac
 - Segregation observed in some early batches suggested addition of spray drying to the procedure, especially for HfB₂ based system
 - Later batches prepared by the milling of mixed powders appeared to be satisfactory
- **Compositions prepared from mixture of ball milled individual powders were unacceptable**
 - Non-homogeneous with large agglomerates and cracks
 - Further milling as mixed powders solved the problem in the one case tried.

ZrB₂ – 20 % SiC Effect of Sintering Time

1h, 2000°C, 10 ksi



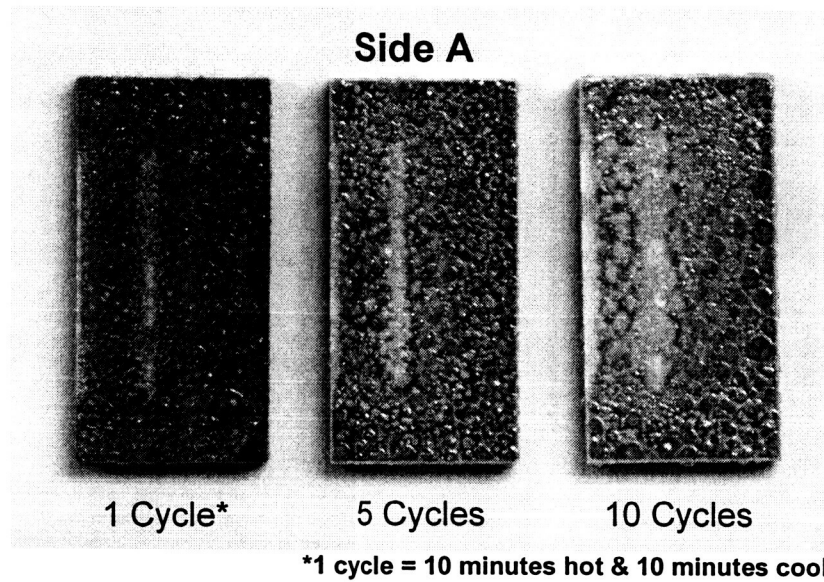
2h, 2000°C, 10 ksi



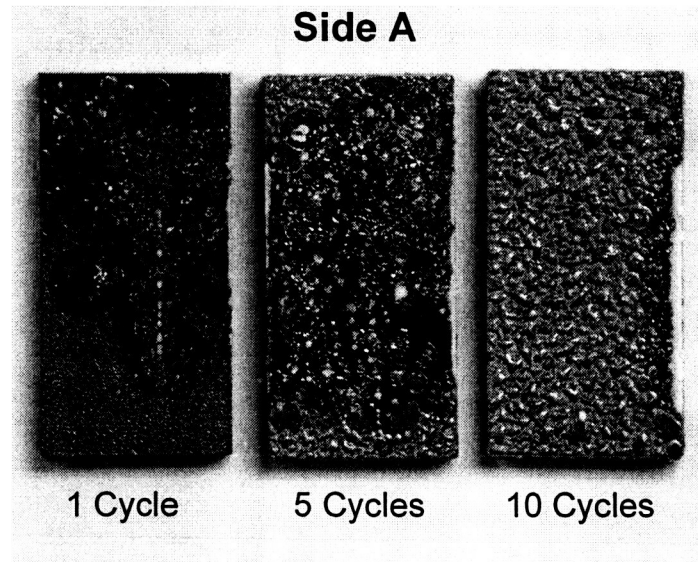
Flexural Strength Results for ZrB₂-based Compositions

Run	Composition	Powder Process	Processing	UTS, Mpa	% strain	E, GPa
877-1	ZrB ₂ -20v/oSiC	batch 1, milled	2000C, 10ksi, 1h, vac	377.4	0.131	289.5
877-2				722.6	0.171	401.5
877-3				749.9	0.181	394.5
877-avg				616.6	0.161	361.8
882-1	ZrB ₂ -20v/oSiC	batch 1, milled	2000C, 10ksi, 2h, vac	381.4	0.086	416.8
882-2				588.8	0.106	531.2
882-3				486.9	0.098	472.3
882-avg				485.7	0.097	473.4
897-1	ZrB ₂ -20v/oSiC	batch 2, milled mtr	2000C, 10ksi, 2h, vac	576.3	0.129	434.3
897-2				560.2	0.133	378.9
897-3				505.2	0.120	395.6
897-avg				547.2	0.127	402.9
878-1	ZrB ₂ -20v/oSiC-20v/o mixed additives	milled mtr	1900C, 10ksi, 2h, vac	838.7	0.178	448.0
878-2				834.3	0.173	475.7
878-3				729.3	0.144	471.1
878-avg				800.8	0.165	464.9
889-1	ZrB ₂ -20v/oSiC-20v/oTaSi ₂	remill mtr milled	1800C, 10ksi, 2h, vac	549.6	0.133	399.0
889-2				786.2	0.185	414.7
889-3				827.0	0.192	424.0
889-avg				720.9	0.170	412.6
908-1	HfB ₂ -20v/oSiC	milled mtr	2000C, 10ksi, 2h, vac	563.8	0.116	473.0
908-2				375.4	0.080	459.6
908-3				518.5	0.110	465.4
908-avg				485.9	0.102	466.0
911-1	HfB ₂ -20v/oSiC	milled mtr	2000C, 10ksi, 2h, vac	549.5	0.118	447.9
911-2				556.0	0.117	453.4
911-3				534.6	0.117	423.4
911-avg				546.7	0.117	441.6

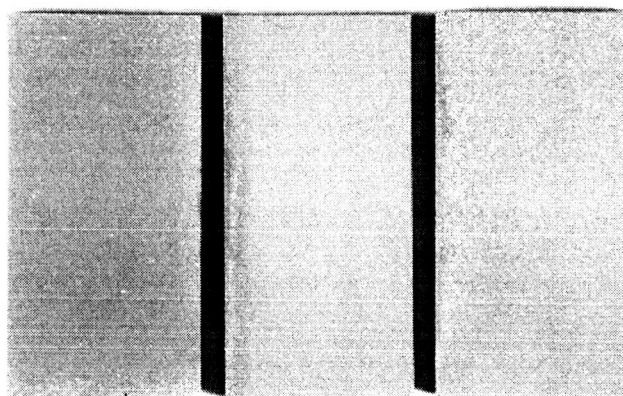
**$\text{ZrB}_2 + 20\% \text{ SiC (ZS)}$ After Furnace
Oxidation at 1627°C**



**$\text{ZrB}_2 + 20\% \text{ SiC} + \text{Mixed Ta Compounds (RB)}$
After Oxidation at 1627°C**



**$\text{ZrB}_2 + 20\% \text{ SiC} + 20\% \text{ TaSi}_2$ (ZSTS)
Oxidized at 1627°C
Side A**

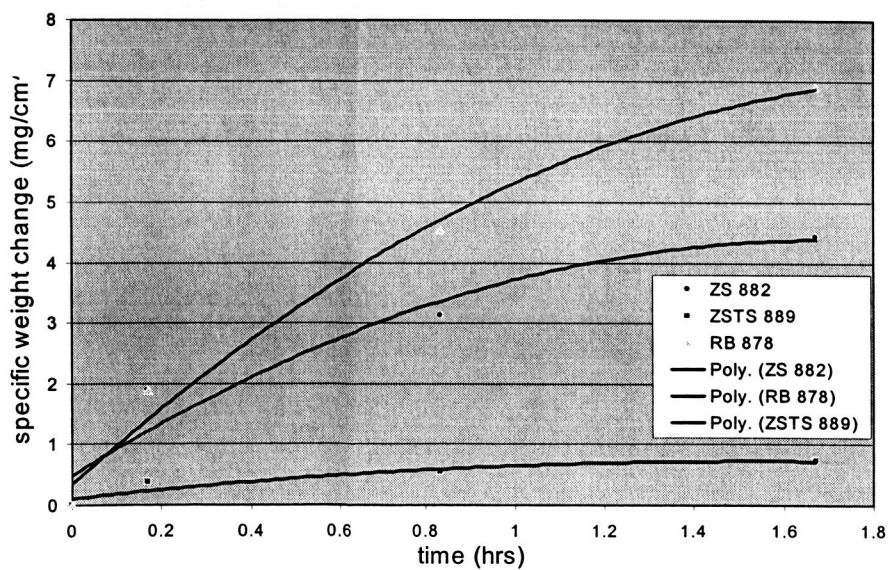


1 Cycle

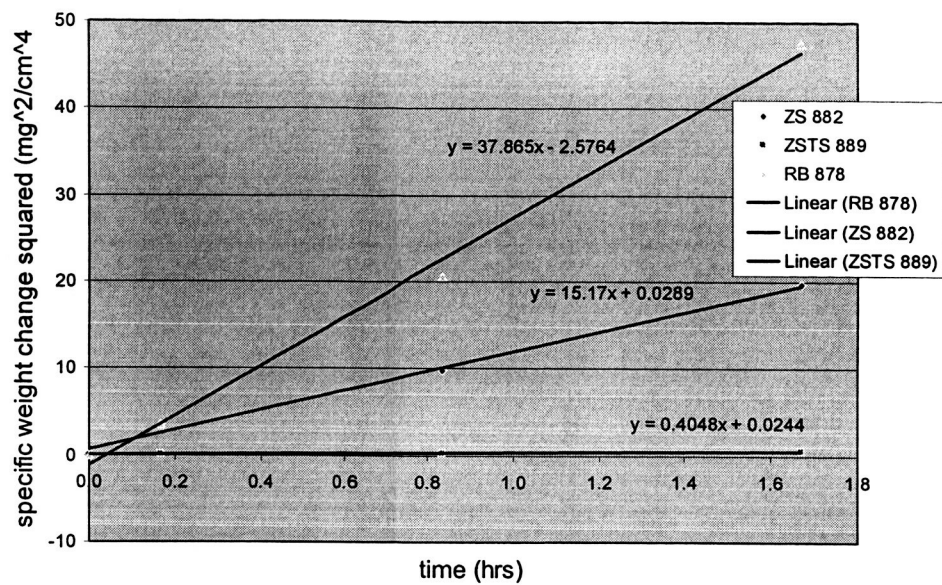
5 Cycles

10 Cycles

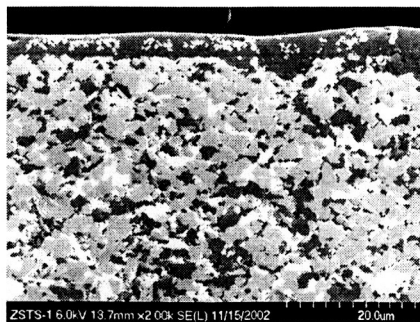
UHTC Oxidation at 1627°C in Zirconia Furnace



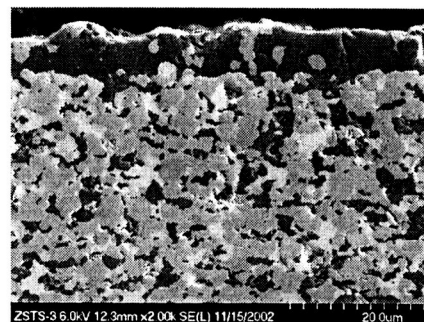
UHTC Oxidation at 1627°C in Zirconia Furnace



20 %TaSi₂ Modified ZrB₂ + 20% SiC After Furnace Oxidation at 1627°C in Air

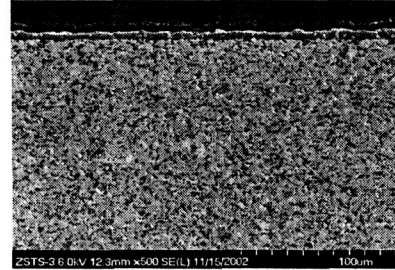
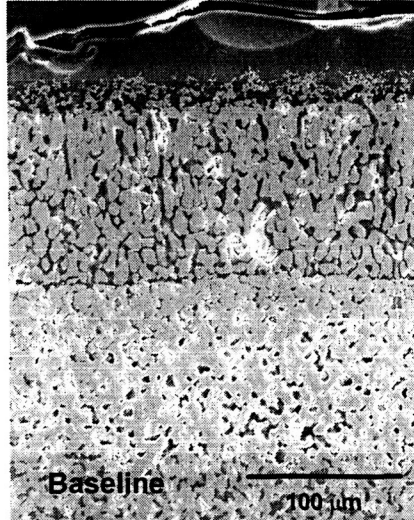


1x10 min cycle, at 1000x



10x10min cycles, at 2000x

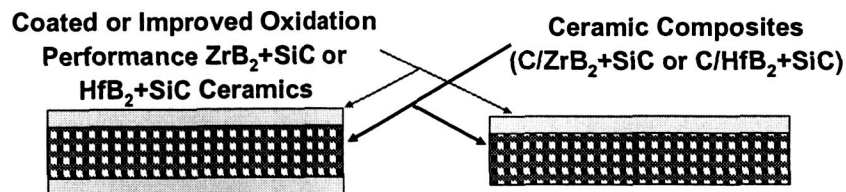
**➤ 10X Improvement in Oxidation
Resistance of TaSi₂ Modified 20 % SiC
10x10 minute cycles, air, 1627°C**



TaSi₂ Modified ZrB₂-20 % SiC

**>10 X improvement in oxidation
resistance relative to industry
baseline based on weight
change and oxidation damage**

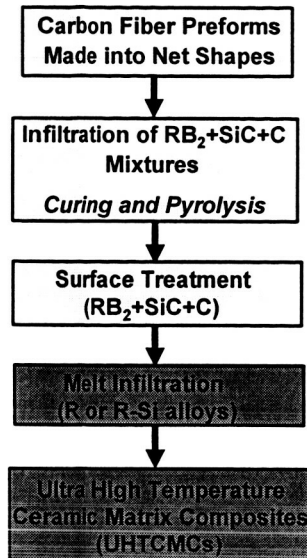
UHTCMC Hybrid System



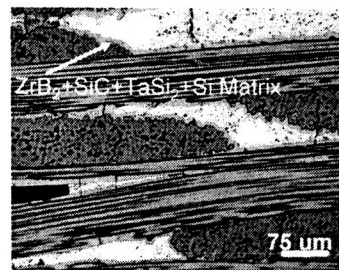
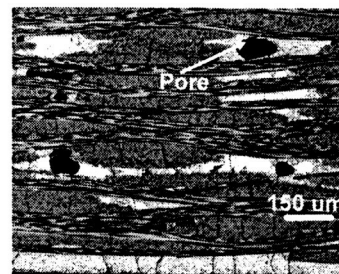
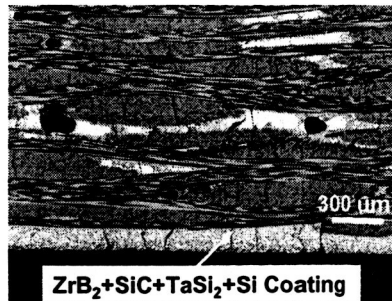
Advantages of UHTCMC hybrids

- Thickness and composition of UHTC layers can be changed to suit the application requirements
- Improved toughness
- Improved environmental durability
 - Tailored surface coatings
 - Crack healing matrices
 - Control of residual stresses
- Smooth composite surfaces
 - Low drag
 - Machinable without fiber damage
 - Easier to bond and attach sensors and other devices
 - Critical to good attachments/seals

UHTCMC Hybrid System Processing

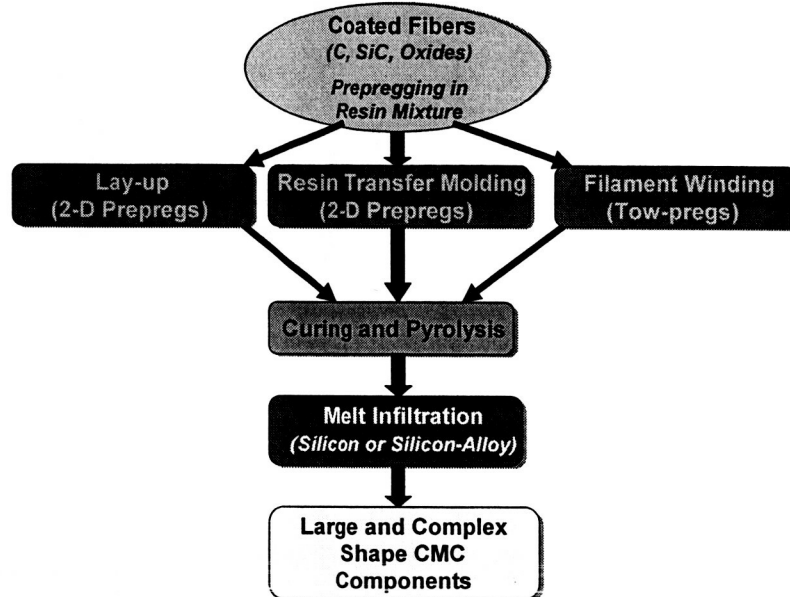


Microstructure of UHTCMC Hybrid System

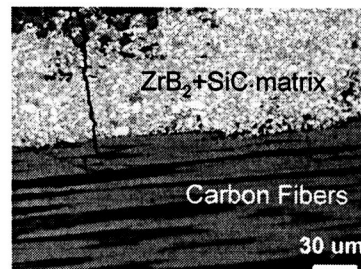
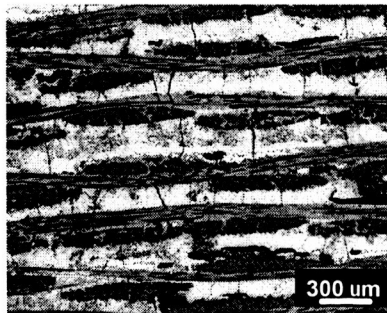


- $\text{ZrB}_2 + \text{SiC}$ slurry infiltrant in C/SiC preform
- Similar microstructures in silicon and Si-Ta alloy melt infiltrated systems
- Cracked coatings and matrix due to thermal expansion mismatch
- Process optimization and property characterization underway

CMCs by Prepregging and Melt Infiltration (PREMI)



Microstructure of UHTCMCs by Prepregging and Melt Infiltration (PREMI)



- 12 layers of T-300, 5-HS cloth prepregged with ZrB₂+SiC or ZrB₂+TaSi₂ mixture
- Warm pressed, pyrolyzed, and melt
- Infiltrated with Si or Si-Ta alloy

Cracking -
All the
pores did
not wst.

Summary

- **Current UHTCs lack robustness (low fracture toughness, reliability and oxidation resistance)**
- **Alloy and functionally graded materials approaches have been identified to improve oxidation resistance**
 - Ta addition appears to be promising with oxidation rate reduced by > 10X @ 1627°C
 - FGM approaches in processing
- **Several composites approaches have been identified to increase mechanical robustness**
 - Infiltration processing and prepregging have produced materials with interesting microstructures
 - Further characterization of microstructures, and mechanical property and environmental durability is needed to guide the next processing cycle

Conclusions

- **Alloying can yield a large improvement in oxidation resistance in a static environment**
 - Need to demonstrate in a flowing environment representative of the application
- **Several approaches to fabrication of UHTC composites appear to be promising in so far as microstructural appearance**
 - Mechanical property and environmental durability evaluations will guide future directions



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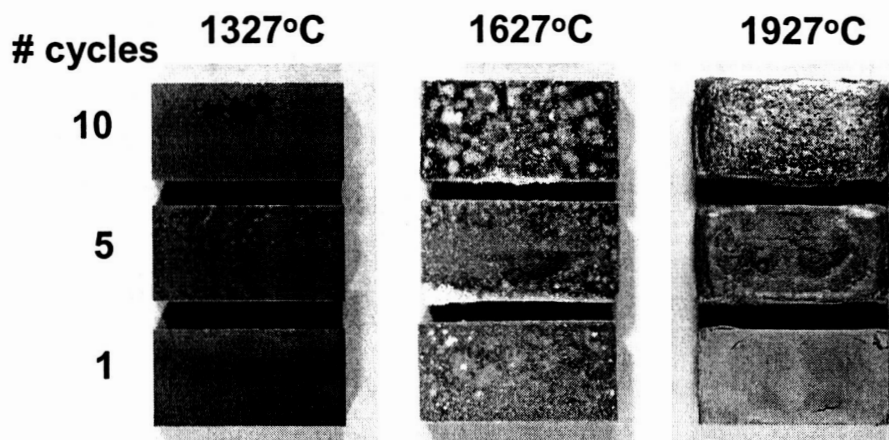
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ZrB₂/ 20 % SiC UHTC Oxidized in Air

10 Minute Cycles



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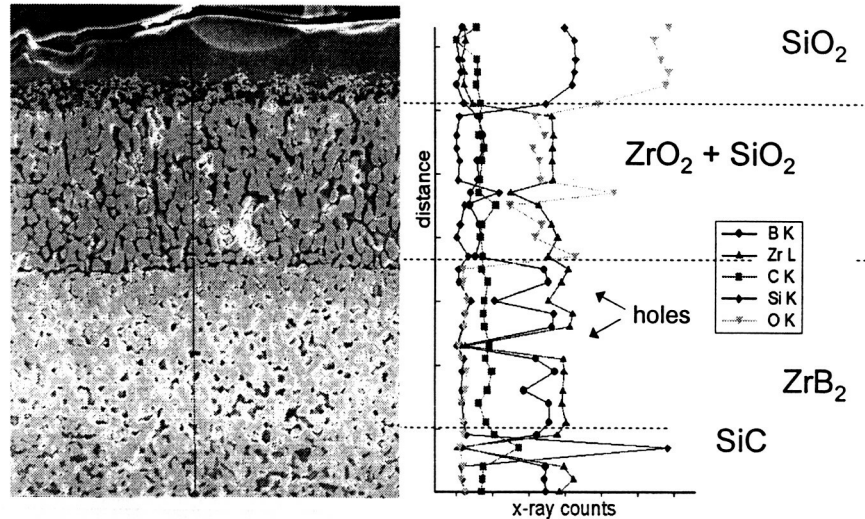
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SEM/EDS: ZrB_2 - SiC 1627°C, Air, Ten 10 min Cycles



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Oxidation Resistance

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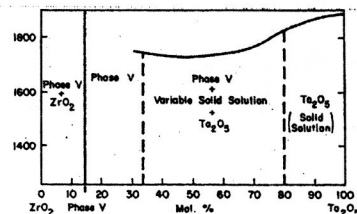
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Mechanisms for ZrO₂ Protective Scale Enhancement via Ta₂O₅ Additions

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- Ta⁺⁵ as a dopant in ZrO₂ lattice
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 - Phase V
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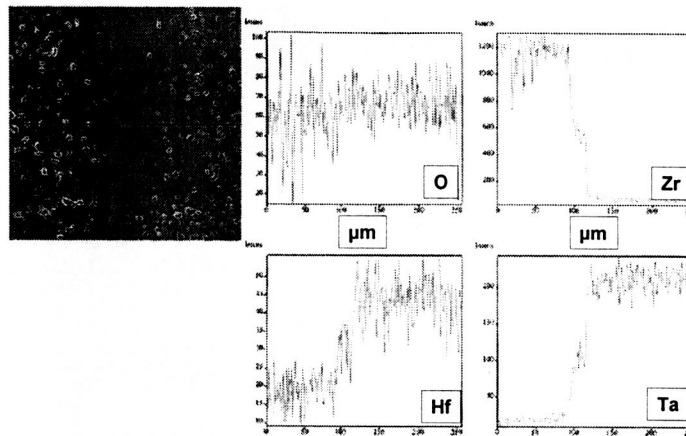
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ZrO₂-Ta₂O₅ Diffusion Couple 1450°C, 1 h



Intermediate phase formation and Ta solubility are evident



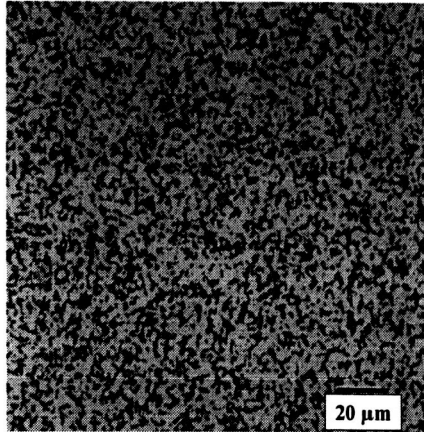
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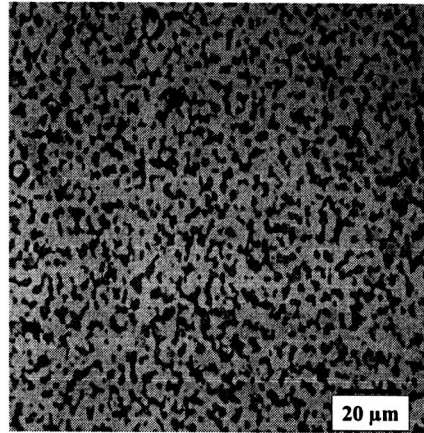


ZrB₂ – 20 % SiC Effect of Sintering Time

1h, 2000°C, 10 ksi



2h, 2000°C, 10 ksi



1/15/2003

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15



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877-3				749.9	0.181	394.5
877-avg				616.6	0.161	361.8
882-1	ZrB ₂ -20v/oSiC	batch 1, mix milled	2000C, 10ksi, 2h, vac	381.4	0.086	416.8
882-2				588.8	0.106	531.2
882-3				486.9	0.098	472.3
882-avg				485.7	0.097	473.4
897-1	ZrB ₂ -20v/oSiC	batch 2, milled mix	2000C, 10ksi, 2h, vac	576.3	0.129	434.3
897-2				560.2	0.133	378.9
897-3				505.2	0.120	395.6
897-avg				547.2	0.127	402.9
878-1	ZrB ₂ -20v/oSiC-20v/o mixed additives	milled mix	1900C, 10ksi, 2h, vac	838.7	0.178	448.0
878-2				834.3	0.173	475.7
878-3				729.3	0.144	471.1
878-avg				800.8	0.165	464.9
889-1	ZrB ₂ -20v/oSiC-20v/oTaSi ₂	remil mix milled	1600C, 10ksi, 2h, vac	549.6	0.133	399.0
889-2				786.2	0.185	414.7
889-3				827.0	0.192	424.0
889-avg				720.9	0.170	412.6
908-1	HfB ₂ -20v/oSiC	milled mix	2000C, 10ksi, 2h, vac	563.8	0.116	473.0
908-2				375.4	0.080	459.6
908-3				518.5	0.110	465.4
908-avg				485.9	0.102	466.0
911-1	HfB ₂ -20v/oSiC	milled mix	2000C, 10ksi, 2h, vac	549.5	0.118	447.9
911-2				556.0	0.117	453.4
911-3				534.6	0.117	423.4
911-avg				546.7	0.117	441.6

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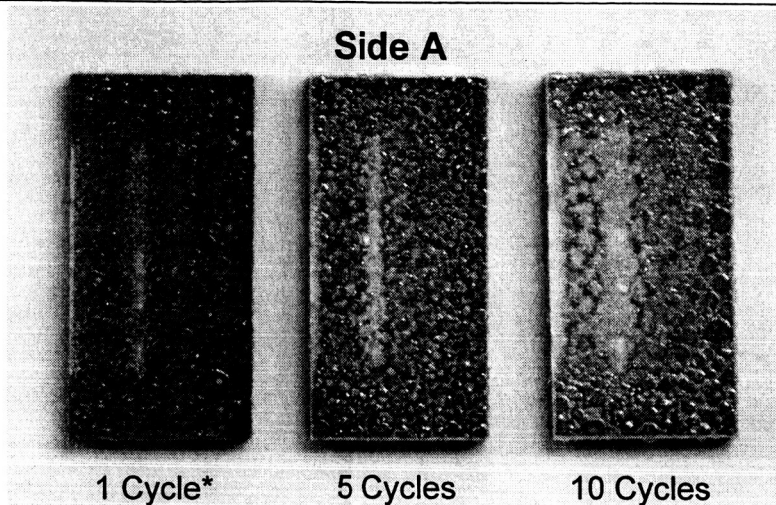
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ZrB₂ + 20 % SiC (ZS) After Furnace Oxidation at 1627°C

Side A



*1 cycle = 10 minutes hot & 10 minutes cool

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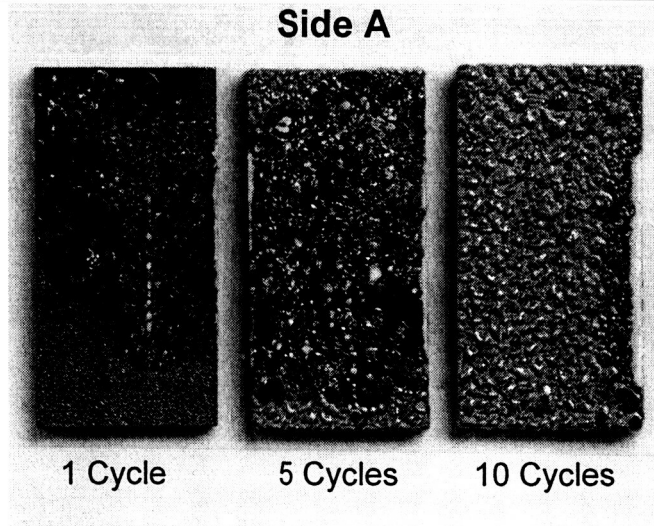
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ZrB₂ + 20 % SiC + Mixed Ta Compounds (RB) After Oxidation at 1627°C

Side A



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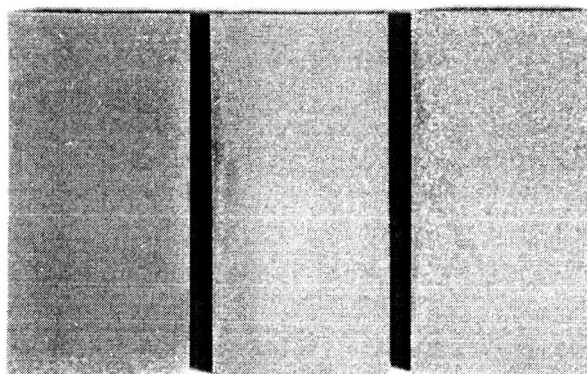
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$\text{ZrB}_2 + 20\% \text{ SiC} + 20\% \text{ TaSi}_2$ (ZSTS) Oxidized at 1627°C

Side A



1 Cycle

5 Cycles

10 Cycles

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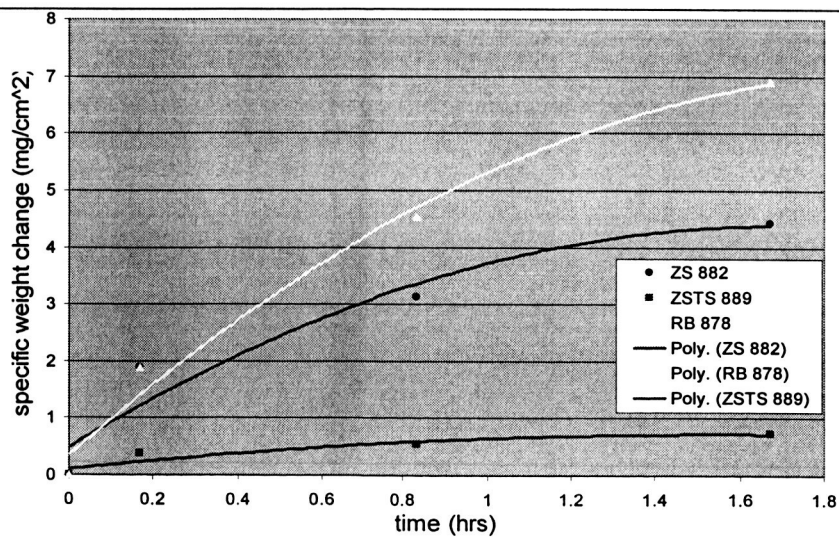
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UHTC Oxidation at 1627°C in Zirconia Furnace



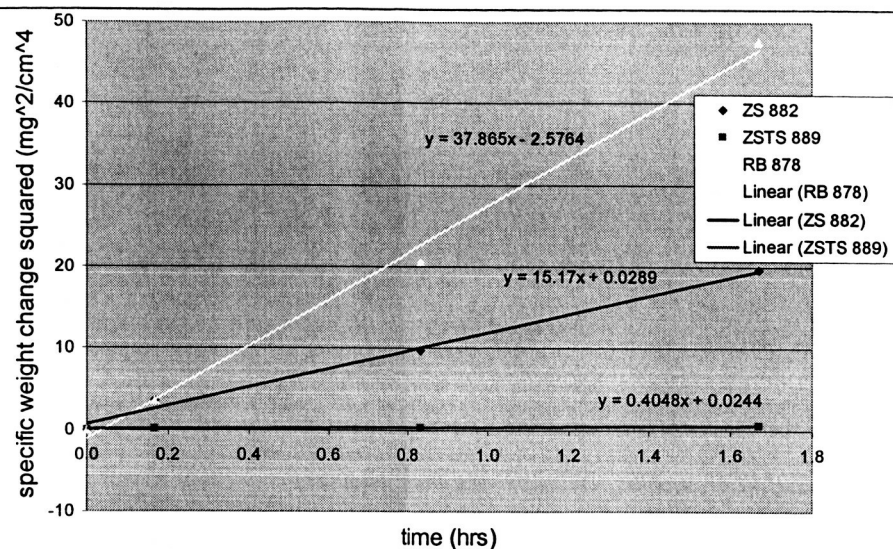
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UHTC Oxidation at 1627°C in Zirconia Furnace



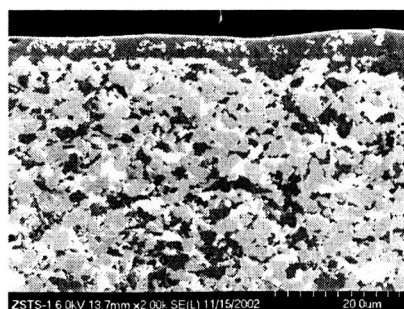
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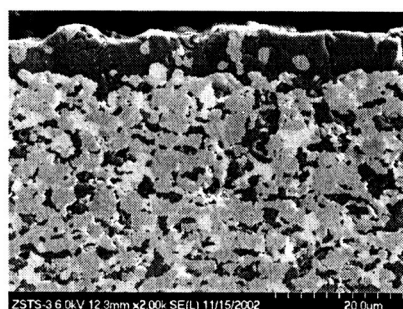
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20 %TaSi₂ Modified ZrB₂ + 20% SiC After Furnace Oxidation at 1627°C in Air



1x10 min cycle, at 1000x



10x10min cycles, at 2000x

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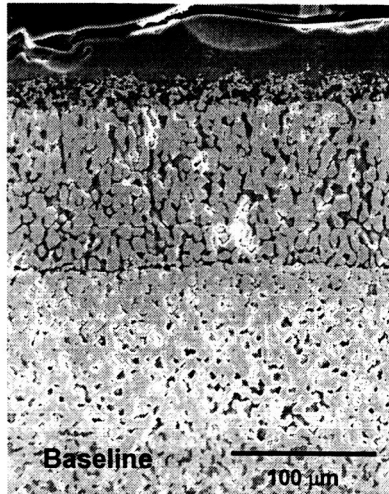
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➤ 10X Improvement in Oxidation Resistance of TaSi_2 Modified 20 % SiC

10x10 minute cycles, air, 1627°C



TaSi_2 Modified ZrB_2 -20 % SiC

**>10 X improvement in oxidation
resistance relative to industry
baseline based on weight
change and oxidation damage**

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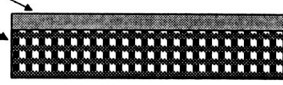
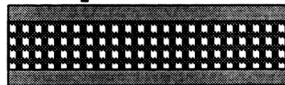


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UHTCMC Hybrid System

Coated or Improved Oxidation
Performance ZrB_2 +SiC or
 HfB_2 +SiC Ceramics

Ceramic Composites
(C/ ZrB_2 +SiC or C/ HfB_2 +SiC)



Advantages of UHTCMC hybrids

- Thickness and composition of UHTC layers can be changed to suit the application requirements
- Improved toughness
- Improved environmental durability
 - Tailored surface coatings
 - Crack healing matrices
 - Control of residual stresses
- Smooth composite surfaces
 - Low drag
 - Machinable without fiber damage
 - Easier to bond and attach sensors and other devices
 - Critical to good attachments/seals

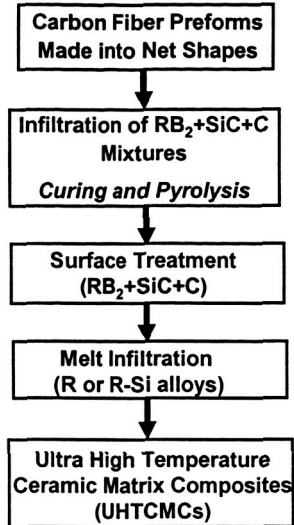
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UHTCMC Hybrid System Processing



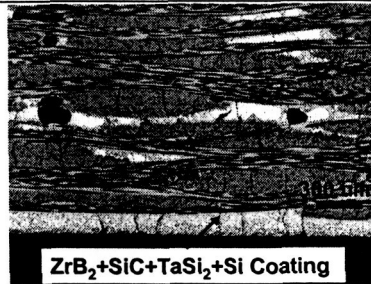
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Microstructure of UHTCMC Hybrid System



- $\text{ZrB}_2 + \text{SiC}$ slurry infiltrant in C/SiC preform
- Similar microstructures in silicon and Si-Ta alloy melt infiltrated systems
- Cracked coatings and matrix due to thermal expansion mismatch
- Process optimization and property characterization underway



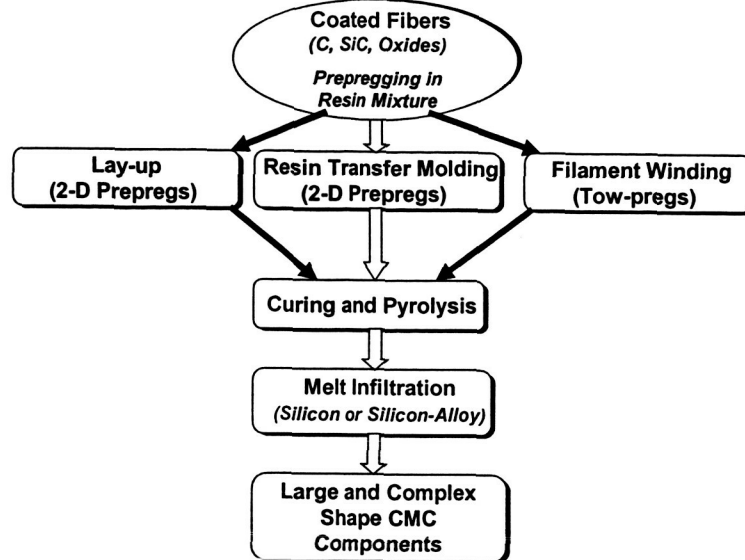
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CMCs by Prepregging and Melt Infiltration (PREMI)



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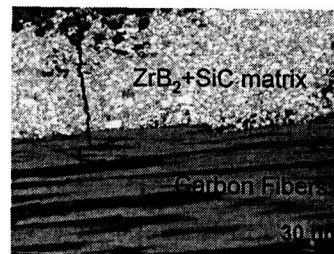
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Microstructure of UHTCMCs by Prepregging and Melt Infiltration (PREMI)



- 12 layers of T-300, 5-HS cloth prepregged with ZrB_2+SiC or ZrB_2+TaSi_2 mixture
- Warm pressed, pyrolyzed, and melt
- Infiltrated with Si or Si-Ta alloy



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Summary

- **Current UHTCs lack robustness (low fracture toughness, reliability and oxidation resistance)**
- **Alloy and functionally graded materials approaches have been identified to improve oxidation resistance**
 - Ta addition appears to be promising with oxidation rate reduced by > 10X @ 1627°C
 - FGM approaches in processing
- **Several composites approaches have been identified to increase mechanical robustness**
 - Infiltration processing and prepregging have produced materials with interesting microstructures
 - Further characterization of microstructures, and mechanical property and environmental durability is needed to guide the next processing cycle



Conclusions

- **Alloying can yield a large improvement in oxidation resistance in a static environment**
 - Need to demonstrate in a flowing environment representative of the application
- **Several approaches to fabrication of UHTC composites appear to be promising in so far as microstructural appearance**
 - Mechanical property and environmental durability evaluations will guide future directions